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B. Weiss

ABSTRACT. Determination of the dislocation structures of two austenitic steels, stabilized by niobium, as dependent on alternate stressing endured in the fatigue and cyclic stress ranges. Electron-optical photographs of the characteristic dislocation arrangements. Comparison of the results with a model conception.

Austenitic steels with 15 to 20% Cr and 10 to 13% Ni are frequently used /155* materials. Despite this fact, no unambiguous results are available concerning their fatigue behavior or the changes occurring in the dislocation structure during the fatigue process [1-3]. From a physical standpoint, the materials have low stacking fault energies. This leads to the fact that transverse slipping is difficult and that the occurrence of dislocation rings, tangles and cells can be expected at high stresses only.

Dislocation structures were found at alternating stress cycle numbers of 10^7 which did not differ greatly from those found in unidirectional deformation. [1]. At stresses leading to fracture after 5×10^4 cycles, the occurrence of numerous dislocation rings was observed [2], but no cell formation. In a more recent electron-optical investigation in samples of a steel with approximately 18% Cr and 13% Ni zones were found, which, depending on stress and number of cycles, were characterized by dislocation pileups, dislocation rings oriented in the [112] direction and by tetrahedrons of stacking faults [3]. At high alternating stress, the occurrence of dislocation cells is also reported.

In the following, changes in the structure of two Nb-stabilized austenitic Cr-Ni steels as a function of the magnitude and extent of exposure to alternating stresses are investigated with the aid of electron microscopy.

Experimental Procedure

The materials used consisted of an austenitic steel made from high-purity

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* Numbers in the margin indicate pagination in the foreign text.

materials and stabilized with Nb, containing roughly 0.1% C, 18% Cr, 12% Ni and 1% Nb (Steel No. 1) and the commercial steel X8CrNiNb 16 13, Material Number 1.4961 with maximum 0.1% C, roughly 16% Cr, 13% Ni and 0.8% Nb (Steel No. 2). Steel 1 was fully austenitic following heat treatment at 1300°C for 4 hours in a quartz capsule filled with Ar and subsequent quenching in water, with an average grain size of 200 μm and a few undissolved Nb carbides. The specimens were threaded, 50 mm long polished round rods. The part of the specimen to be investigated had a length of approximately 10 mm and 3 mm diameter, so that an adequate number of foils could be taken for the electron microscope study. Steel was available in the form of 1.5 mm strips in the shape of tensile specimens. The surface area to be investigated had the dimensions of 20 x 15 mm^2. Heat treatment of the steel was given as 1050 to 1100°C, 20 minutes, air cooling. The fully austenitic structure was fine-grained, with an average grain size of 10 μm and some undissolved Nb carbides with an average size of < 0.5 μm.

The tensile-compression alternating stress was applied in 65 cycles/sec., with a mean stress of 0. Wöhler curves were established for both steels at 20°C. The surface temperature was higher by approximately 10° than that of the environment. At stresses which lead to the failure of the specimens at roughly 3×10^4 cycles, surface temperatures, due to the high damping of austenitic materials [4], rose to the extent that the exposure of the specimens could not be tested.

For the electron microscope examination, 0.4 mm thick platelets were cut with a spark saw from the fatigued specimens. Using a method developed by F. Driver [5], foils were prepared from the platelets by jet polishing with 10% nitric acid and subsequent bath polishing with a solution of 20% glycerine, 10% perchloric acid and 70% ethyl alcohol. To prepare foil from the strips, a method proposed by K. L. Maurer and H. Schaffer [6] was used. Two instruments with 100 kV accleration potential were available for the transmission studies.

Experimental Results

Wöhler curves, presented in Figure 1, were established for both steels. While the yield strengths of the two steels were nearly identical ($\sigma_{0.2\,\text{steel}\,1}$ = 21 kg/mm^2 and $\sigma_{0.2\,\text{steel}\,2}$ = 22 kg/mm^2), the shape of the Wöhler curves /156 differ considerably. The difference may be explained by the different specimen configurations and surface treatments [7, 8].

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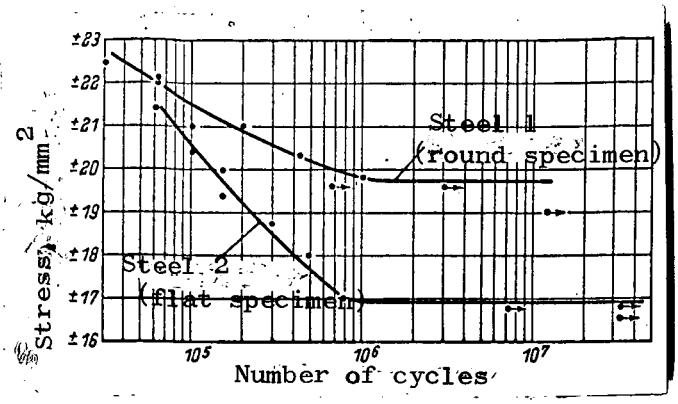

Figure 1. Tensile-compression alternating stress withstood by specimens of two Nb-stabilized austenitic steels as a function of number of cycles.

Figure 2. Dislocation structure of Steel 1 following fatigue exposure within the endurance limit.

As proposed by several researchers [9, 10], the microstructure can be roughly correlated with two ranges of the Wöhler curve: the range of high stress and elongation amplitudes with corresponding number of cycles of $< 10^6$ and the range of low amplitudes with cycle numbers of $> 10^6$. An attempt will be made to qualitatively arrange the dislocation structure obtained of the two steels in this scheme.

For Steel 1 in the stress range of 23 to 20 kg/mm^2, i.e. in the sharply descending part of the stress-cycle curve, similar structures were found. A characteristic arrangement of dislocations is shown in Figure 2. In addition to high dislocation density zones, areas are seen which are nearly free of dislocations. The dislocation tangles, which are difficult to resolve even at high magnifications, consist in essence of dislocations with jumps, dislocation rings and dipoles, oriented in the [112] direction. Heat treatment at 400°C for /157 3 hours leads to the dissolution of the dislocations pileups, which is attributed

to the recovery of individual defect locations and small dislocation rings. As a result, rings oriented in the [112] direction are readily recognized. (Figure 3). The dislocation rings have an average length-to-width ratio of roughly 4 to 1 and their average size (longitudinal) is 500 to 700 Å. Some of the specimens showed a cell structure which was particularly pronounced in the vicinity of the fracture. The average cell size was approximately 1 μm; the cells displayed no preferred orientation. In many cells the walls have a simple geometric structure. (Figure 4).

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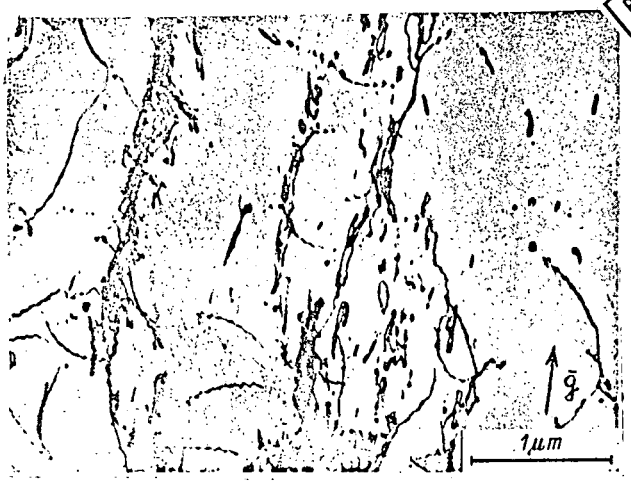

Figure 3. Dislocation rings in a specimen of Steel 1 after fatigue exposure within the range of the endurance limit and subsequent heat treatment of 400°C, 3 hours. Zone axis [111]; $\bar{g} = (\bar{1}\bar{1}2)$.

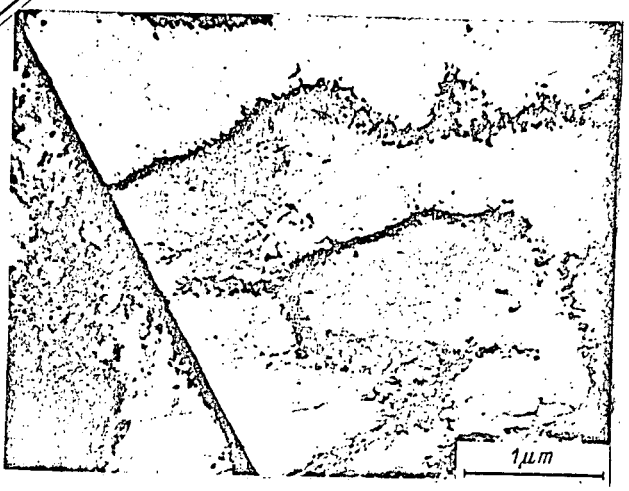

Figure 4. Cell structure in the vicinity of the fracture within the endurance limit of Steel 1.

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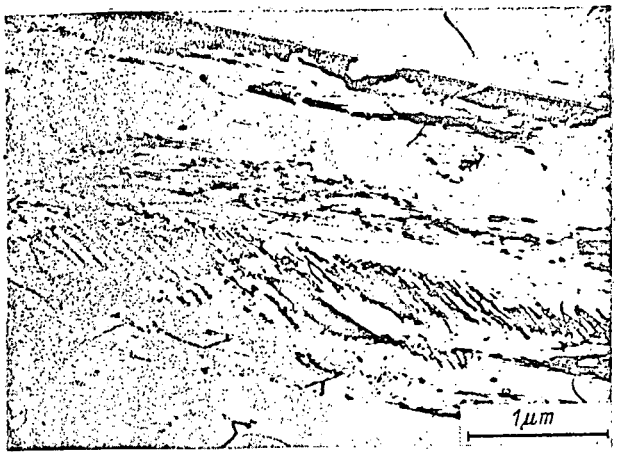

Figure 5. Dislocation structure within the endurance limit of a specimen of Steel 1. Zone Axis [123]; $\bar{g} = (\bar{1}\bar{1}1)$.

Figure 6. Dislocation multipole in a specimen of Steel 1 with visible diagonal Strip Pattern.

With stress amplitudes within the endurance limit of the steel a characteristic dislocation structure is observed, which appears in the form of a stacking fault contrast (Figure 5). These faults are always arranged in a manner parallel to the slip bands. In these slip bands, in a type of diagonal stripe, individual dislocations may be seen, which are parallel to the [112] direction. The occurrence of such faults in unidirectionally deformed austenitic steels has been reported earlier [11, 12]. According to the dynamic theory of electron diffraction [13] these diffraction contrasts represent, in contrast to stacking faults, a dislocation multipole which should be considered a series of dislocations with the same Burgers vector restricted to two parallel slip planes. Assuming that this theory, originally established for multipoles in a unidirectionally deformed aluminum alloy with 10% at. Cu, is valid for other materials with low stacking fault energies also, the following may be cited to distinguish them from

stacking faults: multipoles, compared with stacking faults, yield strip contrast in all low-order reflections. The latter were confirmed for the forms $\{002\}$, $\{\bar{1}11\}$ (Figure 5) and $\{220\}$. In addition, investigations showed that at high magnifications the stripes were less pronounced [13]. Figure 6 indicates that the defect location displays at high magnification a weak diagonal strip pattern in addition to the stacking fault contrast.

Figure 5 also displays the individual dislocations and dipoles, which, if sufficiently removed from each other, yield no strip contrast. For further distinction, the stacking fault contrast can be almost eliminated by lightly tilting the foil, while retaining the orientation (Figure 7a and b).

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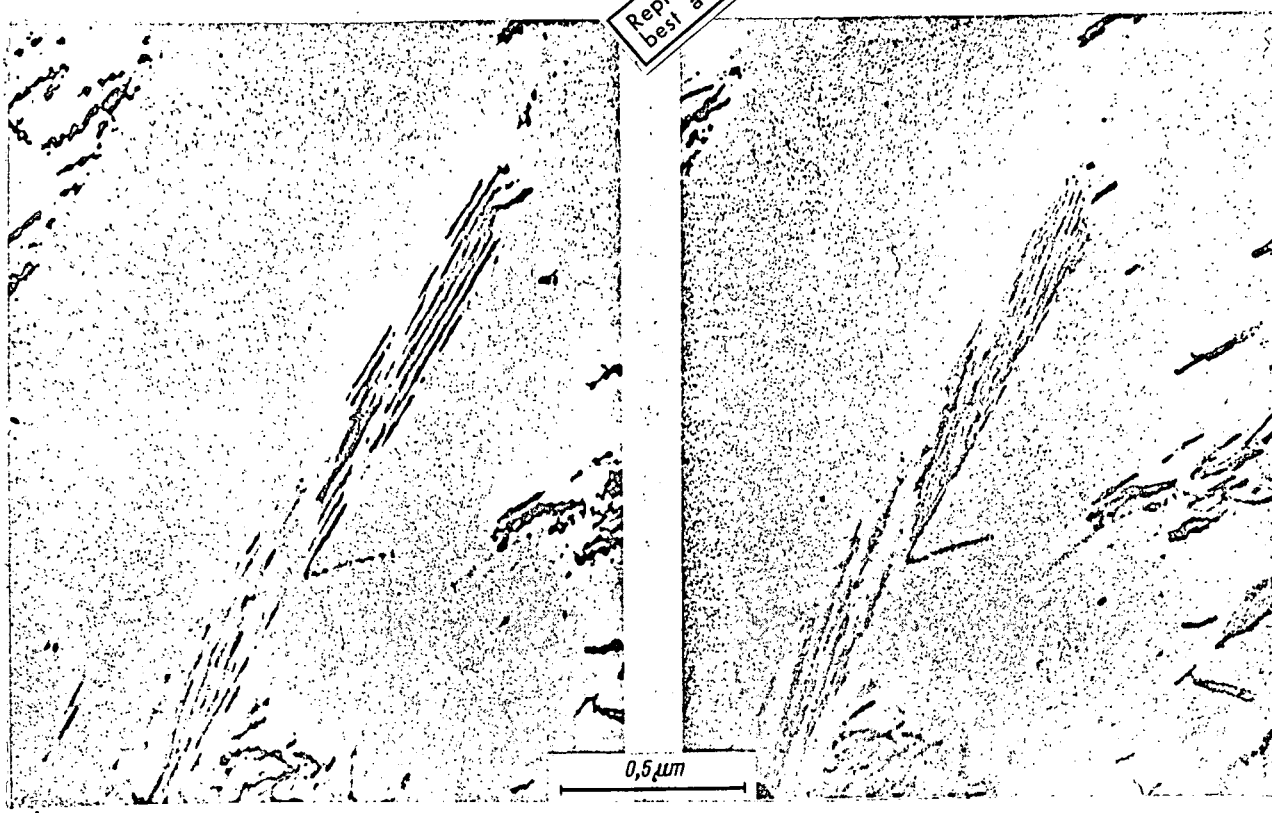

a) Original position of the foil

b) Slightly changed position of the foil in the positive direction.

Figure 7a and b. Effect of a slight tilting of the foil on the strip contrast. Zone Axis [123]; $\bar{g} = (\bar{1}\bar{1}\bar{1})$.

In the corresponding stress range of 21 to 17 kg/mm^2 a similar dislocation structure is obtained for Steel 2 (Figure 8). However, the average dislocation structure is less and the dislocations are seldom arranged in cells. Although here again dislocation tangles and rings are found, the dislocations are present for the most part in the form of bands (Figure 8). In addition, a large number of Nb carbides are visible. Stress within the endurance limit in the case of this steel yield only a slight increase in dislocation density. Individual dislocation rings and multipoles were found, however. Figure 9 indicates that the dislocations here again are arranged in lines.

Discussion of Experimental Results

The dislocation structures of the two steels obtained are readily correlated with the two ranges of the Wöhler curve. In the range of large amplitudes, dislocation patterns, which essentially consist of dislocation rings, dipoles and tangles in a longitudinal arrangement, are found. At high stress cycles, /158
particularly Steel 1 is characterized by a large number of dislocation multipoles. These phenomena fit well into a model recently developed by C. E. Feltner and C. Laird [15, 16]. It was found that the arrangement of dislocations in alternately stressed metals in essence depends on the stacking fault energy and the applied stress. The present investigation again confirms the effect of the stacking fault energy. Measurements yielded a stacking fault energy in a steel comparable to Steel 1 of 15 erg/cm^2 [11]. This low value leads to the fact that, as in the case of unidirectional deformation, the mechanism of transverse conduction is hindered and does not become effective until higher stresses are applied (local temperature rises cannot be excluded) and result in dislocation structures shown in Figure 2 and 8. For the generation of longitudinal dislocation rings the Gilman-Fourie mechanism proposed for alternately stressed aluminum specimens by C. E. Feltner [17] can certainly be assumed. The fact that in addition to transverse conduction, point defects also play a role, which is not entirely clear, may be derived both from the effect of annealing (Figure 3) and from recently performed resistivity measurements on alternately stressed copper specimens [18, 19].

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Figure 8. Dislocation structure of a specimen of Steel 2 after alternate stressing within the endurance limit.

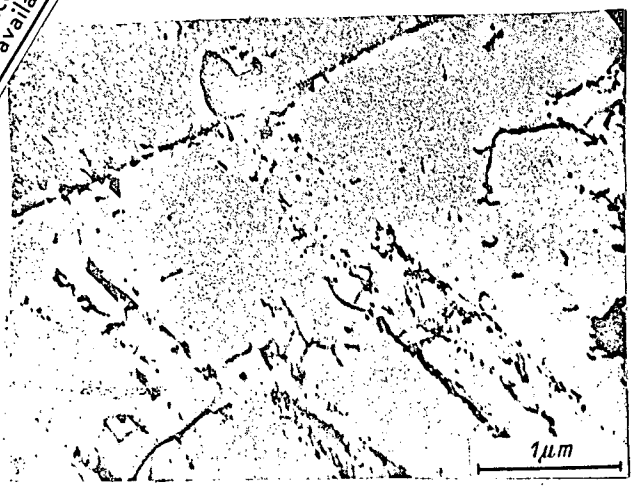

Figure 9. Dislocation structure of a specimen of Steel 2 after alternate stressing within the endurance limit.

Multipoles, are characteristic of metals with low stacking fault energies [15], however, nothing is know concerning their formation by alternate stressing. It may be assumed, however, that as in the case of unidirectional deformation, the dislocations interact on parallel slip planes at suitable distances and thus form a stable arrangement ("trapping mechanism"). If the vertical distance of the individual dislocations is measured, a value of approximately 70 Å is obtained.

To determine the stress which leads to the dissolution of a multipole composed of step dislocations, the following equation has been derived [13]:

$$\tau_p = \frac{G \cdot b}{8\pi(1-\nu)\cdot y} \qquad (1)$$

Here, G is the shear modulus, b the Burgers vector of the step dislocation, ν Poisson's number and y the vertical distance of the individual dislocations. Inserting 1/3 for the Poisson number, 8×10^{11} dyn/cm^2 for the shear modulus and 2.45 Å for the Burgers vector of the effective half [110] dislocation, yields a value of approximately 17 kg/mm^2 for stress. Taking into consideration the experimentally determined value of 20 kg/mm^2 (Steel 1) at which multipoles are dissolved, the type of applied stress and the orientation factor of 1/2, the stress is in agreement with the calculated value. Therefore, the assumptions of P.M. Hazzledine [14] concerning multipole formation after unidirectional deformation

can be applied to alternating stress also. However, defect density is somewhat higher after alternating stressing.

The investigation showed that the arrangement of dislocations in Steel 1 and 2, especially with respect to multipole formation, is affected in addition to stacking fault energy and stress, by other factors, such as grain size and impurity content.

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Summary

Stress cycles to failure were measured on round specimen of austenitic steel melted in the laboratory with approximately 0.1% C, 18% Cr, 12% Ni and 1% Nb and a commercial steel with maximum 0.1% C, 16% Cr, 13% Ni and 0.8% Nb as a function of applied tension-compression alternating stress. Electron optical studies were performed on the failed specimens to determine the dislocation structure. The dislocation arrangements may be correlated with the two indicated ranges of the Wöhler curve; at high stress amplitudes dislocation tangles, dislocation rings arranged in a longitudinal [112] direction and dipoles and at high cycle numbers, dislocation multipoles, are found.

The results fit well the model of Feltner and Laird connecting the effect of stacking fault energy and stress amplitude on dislocation arrangements in alternately stressed metals.

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